

## PATENT SPECIFICATION

1,169,538



1,169,5

NO DRAWINGS

Inventors: ERNST CONRAD and GONAN FROSTELL

Date of filing Complete Specification: 24 Nov. 1966.

No. 50609/65.

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Int. Cl.:—C 13 I 1/08

## COMPLETE SPECIFICATION

## Sugar Substitute and a Process for the Preparation thereof

W. LYCKEBY STARKESEFORADLING AB, a mixture undergoing hydrogenation are hydrogenated substantially without any reduction of units

ERRATUMSPECIFICATION NO. 1,169,538

Page 3, line 34, after "minutes" insert "(or the 15 minutes according to Example 2"

THE PATENT OFFICE,  
27 November 1969

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actions on teeth.

However, it has been established that the consumption of sorbitol-containing food-stuffs or candies may sometimes cause 20 digestion troubles on account of the small resorption of sorbitol in the intestines, the result being a laxative action. In sensitive persons said troubles begin after consumption of about 50 grams of pure sorbitol; other 25 persons may tolerate a considerably greater amount without any side effects occurring.

From the above statements it is obvious that there is a need for a further improved 30 substitute for sugar for use in candies, confitures and similar products, which substitute does not cause any digestion troubles and decreases the risk of the formation of caries in teeth.

The present invention relates to a process 35 for preparing an improved sugar substitute which has no laxative action and does not give rise to caries attack on teeth or only in a negligible extent.

According to the present invention a 40 process for preparing a sugar substitute comprises so hydrogenating a mixture of dextrines and other poly- and oligo-saccharides having a dextrose equivalent value of from 1.2 to less than 15%, obtained 45 by a process including the step of hydrolysing starch, that all reducing groups present in the

only formed in small amounts. The average number of monosaccharide units in the hydrolysis product is from 3 to 5, e.g. 4.

Examples of starches which may be used in the hydrolysis include corn starch and potato starch.

The hydrolysis may be carried out under acid, weakly hydrolysing conditions e.g. at a pH of 2 to 4 and may be conducted at an elevated temperature e.g. 150°C. A weak organic acid or an inorganic acid or an acidic salt may be used to provide the desired pH. The hydrolysis temperature, pH and pressure are chosen so as to give the desired partial hydrolysis.

The hydrolysis is preferably carried out by use of a weakly active enzyme, the hydrolysis being performed under the optimum conditions for the particular enzyme used. Many suitable enzymes are commercially available, one very satisfactory enzyme for the purpose being "alpha-Amylase" sold by Novo A/S of Copenhagen, Denmark.

The hydrolysis is stopped when the product has the desired dextrose equivalent value. When the hydrolysis is an acid hydrolysis carried out at an elevated temperature it may be halted by neutralization of the acid and/or by decreasing the temperature whilst if the hydrolysis is

[Price]

SEE ERRATA SLIP ATTACHED

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## COMPLETE SPECIFICATION

### Sugar Substitute and a Process for the Preparation thereof

We, LYCKEBY STARKELESEFORADLING AB, a Swedish joint stock company, residing at Lyckeby, Sweden, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:

In order to prevent caries it is known to use sorbitol for the preparation of all kinds of sweets and candies which usually contain sugar. The advantage in using sorbitol is that it does not cause fermentation which is regarded as the basic reason for the formation of caries and other unfavourable actions on teeth.

However, it has been established that the consumption of sorbitol-containing food-stuffs or candies may sometimes cause digestion troubles on account of the small resorption of sorbitol in the intestines, the result being a laxative action. In sensitive persons said troubles begin after consumption of about 50 grams of pure sorbitol; other persons may tolerate a considerably greater amount without any side effects occurring.

From the above statements it is obvious that there is a need for a further improved substitute for sugar for use in candies, confitures and similar products, which substitute does not cause any digestion troubles and decreases the risk of the formation of caries in teeth.

The present invention relates to a process for preparing an improved sugar substitute which has no laxative action and does not give rise to caries attack on teeth or only in a negligible extent.

According to the present invention a process for preparing a sugar substitute comprises so hydrogenating a mixture of dextrines and other poly- and oligo-saccharides having a dextrose equivalent value of from 1.2 to less than 15%, obtained by a process including the step of hydrolysing starch, that all reducing groups present in the

mixture undergoing hydrogenation are hydrogenated substantially without any reduction in the number of monosaccharide units per molecule i.e. substantially without any change in ratios between the amounts of saccharides having different numbers of monosaccharide units.

The step of hydrolysing starch is, of course, a partial hydrolysis since the hydrolysis product comprises dextrines and other poly- and oligo-saccharides. The dextrines and other poly- and oligo-saccharides usually contain up to 20 monosaccharide units, preferably 3 to 10 monosaccharide units. Free monosaccharides, e.g. glucose, and disaccharides are only formed in small amounts. Preferably the average number of monosaccharide units in the hydrolysis product is from 3 to 5, e.g. 4.

Examples of starches which may be used in the hydrolysis include corn starch and potato starch.

The hydrolysis may be carried out under acid, weakly hydrolysing conditions e.g. at a pH of 2 to 4 and may be conducted at an elevated temperature e.g. 150°C. A weak organic acid or an inorganic acid or an acidic salt may be used to provide the desired pH. The hydrolysis temperature, pH and pressure are chosen so as to give the desired partial hydrolysis.

The hydrolysis is preferably carried out by use of a weakly active enzyme, the hydrolysis being performed under the optimum conditions for the particular enzyme used. Many suitable enzymes are commercially available, one very satisfactory enzyme for the purpose being "alpha-Amylase" sold by Novo A/S of Copenhagen, Denmark.

The hydrolysis is stopped when the product has the desired dextrose equivalent value. When the hydrolysis is an acid hydrolysis carried out an elevated temperature it may be halted by neutralization of the acid and/or by decreasing the temperature whilst if the hydrolysis is

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enzymatic it can be stopped by increasing the temperature so that the enzyme is destroyed.

If the hydrolysis product is to be hydrogenated directly the hydrolysis is halted before the hydrolysis product has a dextrose equivalent value of 15%. However the hydrolysis product may be subjected to a fermentation step whereby mono- and di-saccharides are removed, the product remaining after the fermentation consisting of a mixture of dextrines and poly- and oligo saccharides having a dextrose equivalent value of at least 1.2% but less than 15% and the said product then hydrogenated. In this case the dextrose equivalent value of the hydrolysis product may exceed 15% as the fermentation step used reduces the dextrose equivalent value to below 15%.

It will be appreciated that critical control of the hydrolysis conditions will often be necessary in order to ensure that only the desired degree of hydrolysis occurs. This control is well within the competence of an expert in this art and the expert in this art is also, of course, competent to determine the dextrose equivalent value of the hydrolysis product and of the product remaining after the fermentation, if a fermentation step is used.

In order to determine the conditions necessary in any particular process it will generally be necessary to conduct a few preliminary tests. For example if in one such test it is found that the hydrolysis conditions used result in the hydrolysis going too far then clearly the hydrolysis conditions are too strong. Another test then has to be conducted using milder hydrolysis conditions or shorter hydrolysis times to see if the desired degree of hydrolysis then occurs.

The hydrolysis product, or the product remaining after the fermentation, if a fermentation step is used, is hydrogenated in a known manner, the conditions being such that all reducing groups present in the mixture undergoing hydrogenation are hydrogenated substantially without any reduction of the number of monosaccharide units per molecule. Thus, the hydrogenation step results in substantially no hydrolysis of the hydrolysis product or of the product remaining after the fermentation step, if a fermentation step is used. The hydrogenation can conveniently be carried out at an elevated temperature and using e.g. Raney nickel as the catalyst.

The hydrogenation serves to provide a product which is considerably less fermentable than the material which is hydrogenated because any reducing groups present in this material are hydrogenated to hydroxy groups.

The sugar substitute obtained by the hydrogenation has a lower degree of sweetness than saccharose, which is due to

the fact that mono or disaccharides have only been formed to a minor extent in the hydrolysis step or have been removed in the optional fermentation step. For this reason, if desired, the product obtained by the hydrogenation step can be admixed with an artificial sweetening agent such as saccharine or sodium cyclamate, preferably in an amount which gives a degree of sweetness corresponding that of saccharose. The result is that the mixture obtained can directly replace a corresponding amount of saccharose in the preparation of food products of many kinds including candies and sweets etc.

The product of the process according to the invention can be prepared in the form of a viscous syrup, i.e. in solution, but it is also possible to subject the solution to a drying step in order to obtain the product in the form of a white powder which is hygroscopic only to a very minor extent. The drying may be performed in any suitable manner, such as spray drying, roller drying or freeze drying.

The invention is further elucidated by the following specific examples, in which parts and per cent refer to the weight of the dry substance, if not otherwise stated, and in which the temperatures are in degrees Celsius.

#### Example 1. Acid hydrolysis.

A suspension of potato starch containing 45% of dry solids (2,000 kgs. of potato flour containing 1,800 kgs. of dry starch suspended in 2,000 litres of water) was admixed with 5 litres of 37% technical hydrochloric acid. The pH of the mixture was 2.2. The hydrolysis was performed in 30 minutes at 130° and at a gauge pressure of 3 at. in order to transform the starch to a dextrinous decomposition product. The amount of reducing substances, calculated as dextrose, was 12%. The batch was neutralized with 20 litres of 15% sodium carbonate solution to a pH of 6.5. The solution was decolourized by the use of active carbon and solid substances were removed by centrifugation. Then Raney nickel catalyst was added to the hydrolysis product in an amount of 2%, i.e. 36 kgs. The hydrolysis product was then hydrogenated at a hydrogen pressure of 75 at. and at a temperature of 145°. The hydrogen consumption was 30 cubic metres hydrogen at NTP. The hydrogenation was performed conventionally and was continued until an equilibrium was reached, that is until all reducing substances were hydrogenated. The catalyst was then removed by separation.

The solution was passed through a cationic and an anionic ion exchange resin for removal of metal and acid ions. The solution was then concentrated by vacuum evaporation and spray dried to yield a non-hygroscopic powder.

*Example 2. Enzymatic hydrolysis.*

The 45% starch suspension according to Example 1 was admixed with 3 kgs. of enzyme (bacterial alpha-Amylase supplied by Novo A/S, Copenhagen, Denmark). The pH of the solution was 6.2. By the addition of sodium carbonate solution the pH was adjusted to 7.5. The suspension was heated for 15 minutes to a temperature of 90°. Then the enzyme was inactivated by heating for 5 minutes at a temperature of 110°. The end product was a solution containing mainly dextrineous decomposition products. The content of reducing substances, calculated as dextrose, was 4.6%. The solution was decolourised and separated and hydrogenated as described in Example 1. In the hydrogenation step the hydrogen consumption was 26 cubic metres at NTP.

When it is desired to obtain a product which can be used as a direct substitute for saccharose the concentrated solution is admixed with sodium cyclamate in an amount corresponding to about 10 grams per kg. dry substance before the spray drying step.

*Example 3. Intermediary fermentation of mono and disaccharides.*

In order to obtain the lowest possible amount of mono and disaccharides in the final product the following intermediary step was used in the process according to Example 1. By increasing the hydrolysis time in Example 1 from 30 minutes to 60 minutes to 60 minutes) a reducing (dextrose equivalent) value of 27% was obtained. The solution was neutralized by the use of 18 litres of a 15% sodium carbonate solution to give a pH of 5.0. The temperature was adjusted to 30° and 10 kgs. of brewer's yeast (*Saccharomyces cerevisiae*) were added. The fermentation was carried out conventionally and was finished after about 48 hours when the formation of carbon dioxide was ended. The amount of reducing sugars had then been decreased to give a dextrose equivalent value of about 1.2%. The solution was decolourised by the use of active carbon and filtered. The clear solution obtained was hydrogenated as previously described. The amount of hydrogen used was 12 cubic metres at NTP. The hydrogenated solution was separated

from the catalyst, filtered and treated with ion exchange resins. In the subsequent evaporation step the alcohol formed during fermentation was removed.

## WHAT WE CLAIM IS:—

1. A process for the preparation of a sugar substitute comprising so hydrogenating a mixture of dextrines and other poly- and oligo-saccharides having a dextrose equivalent value of from 1.2 to less than 15%, obtained by a process including the step of hydrolysing starch, that all reducing groups present in the mixture undergoing hydrogenation are hydrogenated substantially without any reduction in the number of monosaccharide units per molecule.

A process according to claim 1 in which starch is hydrolysed to give the mixture having a dextrose equivalent value of from 1.2 to less than 15% and the product so obtained is subjected to the hydrogenation.

3. A process according to claim 1 in which starch is hydrolysed to give a mixture of dextrines and other poly- and oligo-saccharides having a dextrose equivalent value of at least 1.2%, the product of the hydrolysis is subjected to a fermentation step whereby mono- and di-saccharides are removed, the product of which step is a mixture of dextrines and other poly- and oligo-saccharides having a dextrose equivalent value of from 1.2 to 15%, and the product remaining after the fermentation step is then subjected to the hydrogenation.

4. A process according to any preceding claim in which the starch is hydrolysed by use of an enzyme.

5. A process according to any preceding claim comprising adding an artificial sweetening agent to the hydrogenation product.

6. A process according to claim 1 substantially as described herein with reference to any of the Examples.

7. A sugar substitute prepared by a process according to any preceding claim.

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